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CANDIDATE PROTECTANTS FOR WHEAT AGAINST STORED-GRAIN INSECTS



UNITED STATES
DEPARTMENT OF
AGRICULTURE

MARKETING
RESEARCH REPORT
NUMBER 1080

PREPARED BY
AGRICULTURAL
RESEARCH
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Agricultural Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

Washington, D.C.

December 1977

ABSTRACT

LaHue, D. W. and E. B. Dicke. Candidate protectants for wheat against stored-grain insects. U.S. Department of Agriculture Marketing Research Report No. 1080, 16 pp. 13 tables.

Candidate insecticidal materials were tested on Hard Winter wheat stored in small, intermediate-type bins. A diatomaceous earth dust impregnated with malathion gave the best protection and imparted repellency to the wheat against rice weevils, but addition of the dust reduced the test weight of the wheat.

Of the candidate sprays, pirimiphos-methyl and chlorpyrifos-methyl were superior to the standard dose of malathion in protecting the wheat against attack by a number of species of stored-grain insects. Fenitrothion was generally more effective than malathion.

Key words: Chlorpyrifos-methyl, diatomaceous earth, fenitrothion, grain protectants, insecticides, malathion, pirimiphos-methyl, stored-grain insects, stored-product insects, wheat, wheat protectants.

This publication reports research involving pesticides. It does not contain recommendations for their use, nor does it imply that the uses discussed here have been registered. All uses of pesticides must be registered by appropriate State and/or Federal agencies before they can be recommended.

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Candidate Protectants For Wheat Against Stored-Grain Insects

Delmon W. LaHue and Edwin B. Dicke¹

The candidate insecticidal materials were tested as protectants on wheat stored in small, intermediate-type bins. Damaging infestations of mixed populations of stored-grain insects were established in all bins of untreated (check) wheat during the first 3 months of storage. At the doses applied, pirimiphos-methyl (*O*-[2-diethylamino]-6-methyl-4-pyrimidinyl] *O,O*-dimethyl phosphorothioate) and chlorpyrifos-methyl (*O,O*-dimethyl *O*-[3,5,6-trichloro-2-pyridyl] phosphorothioate) sprays and malathion-Kenite² 2-I (M + K) dust gave excellent protection for 12 months. Fenitrothion (*O,O*-dimethyl *O*-[4-nitro-*m*-tolyl] phosphorothioate) gave good protection during the 12 months' study but did not prevent the establishment of an indigenous

infestation of mixed species of insects during the last month of storage.

The malathion-diatomaceous earth dust (M + K) treatment imparted repellency to the wheat and gave the best control of all four test insect species used in the toxicity studies. Pirimiphos-methyl and chlorpyrifos-methyl treatments were effective against the flour beetles and rice weevils, but some damage occurred in the tests with the lesser grain borers. Fenitrothion was generally more effective than malathion.

Malathion, fenitrothion, and chlorpyrifos-methyl residues³ degraded gradually at about the same rate during the 12 months' storage. Pirimiphos-methyl residues degraded slowly in a somewhat erratic pattern.

BACKGROUND AND OBJECTIVES

Three phases of testing are generally required to develop a protective treatment for grain: (1) preliminary laboratory studies to determine the toxicity and repellency of a test material to stored-product insects, (2) an intermediate evaluation in small bins to compare promising materials at selected dosages with an accepted or standard insecticidal application and with untreated grain, and (3) field-scale bin, warehouse, and elevator tests. The most promising materials are selected from the preliminary laboratory studies for evaluating dosage rates, residue degradation, and efficacy of residual protection in small bin, intermediate-type

storage studies. The 0.14 m³ (5 ft³) cylindrical bins have been extensively used in intermediate-type storage studies with corn (3),⁴ farmers stock peanuts (7), wheat (2,8), and sorghum (4).

Grain protectants must be easy to apply, safe to humans, effective in initial and residual action, and low in cost. Only a few materials that meet these criteria have been approved for use, but the search for additional new and promising residual protectant materials continues. Furthermore, resistance of stored-product insects to insecticides and fumigants has been reported for many years from widespread areas, emphasizing the need for other new, acceptable insecticidal materials. Brown (1) reported that more than 150 cases of acquired insect resistance or tolerance to insecticides were known. However, relatively few of these were stored-product insects.

¹Entomologist and research technician, U.S. Grain Marketing Research Center (USGMRC), Agricultural Research Service, U.S. Department of Agriculture, 1515 College Avenue, Manhattan, Kans. 66502

²Trade names are used in this publication solely to provide specific information. Mention of a trade name does not constitute a warranty of the product by the U.S. Department of Agriculture nor an endorsement over other products not mentioned.

³Loren I. Davidson, physical science technician, USGMRC, made the residue determinations.

⁴Numbers in parentheses refer to items in Literature Cited p. 11.

Parkin and Forster (12) found that red flour beetles, *Tribolium castaneum* (Herbst), from a field-collected culture were far more difficult to kill with malathion than were a laboratory strain. Parkin and Forster (11) and McDougall (9) reported that certain field-collected cultures of the rice weevil, *Sitophilus oryzae* (L.), were more difficult to kill with lindane than laboratory-reared cultures. The probability of such field-acquired resistance or tolerance is of major importance. Laboratory-induced tolerance ratios do not indicate conditions in farm and commercial storages but do indicate what may happen under certain insecticidal selection pressures. The review of reports of resistance by Parkin (10) indicates the need to continue the search for new insecticides.

Pirimiphos-methyl was found to be a promising short-term insecticidal material for controlling the larval forms of the Indian meal moth, *Plodia interpunctella* (Hübner); and, of particular interest, malathion-resistant and nonresistant strains were controlled with the same applications (5). Pirimiphos-methyl, applied as water emulsions on shelled corn and Hard Winter wheat, effectively controlled rice weevils, red flour beetles, and confused flour beetles, *T. confusum* Jacquelin duVal. It also prevented progeny damage for 3 months, but some lesser grain borer (*Rhyzopertha dominica* (F.)) damage occurred at doses of 5 and 10 p/m AI (active ingredient) (6). Zettler (13) reported that five

malathion-resistant strains of the Indian meal moth showed no cross-resistance to pirimiphos-methyl and concluded that pirimiphos-methyl could be a potential replacement for malathion as an insecticidal treatment to protect certain commodities against infestation of this insect species.

Fenitrothion was found to be a promising insecticidal material for controlling three species of *Sitophilus* and two species of *Tribolium* in laboratory tests conducted by the authors. In those tests the efficacy of fenitrothion compared favorably with malathion at equal dosages (5, 7.5, and 10 p/m) during a 12-month study.

Chlorpyrifos-methyl (formulation M-3721) gave excellent control of rice weevils, maize weevils (*Sitophilus zeamais* Motschulsky), red flour beetles, confused flour beetles, and lesser grain borers in other laboratory tests with dosages of 4, 5, 6, 8, and 10 p/m.

The primary objectives of the test reported here, which was conducted from March 1973 to June 1975, were to compare the relative effectiveness of fenitrothion at a dosage of 8.3 p/m AI, chlorpyrifos-methyl (formulation M-3721) at 6.3 p/m AI, pirimiphos-methyl (formulation JF2764) at 7.8 p/m AI, and a candidate malathion-Kenite 2-I (M + K) dust formulation (10.4 p/m AI malathion) with the standard dosage of 10.4 p/m malathion AI in an emulsion on Hard Winter wheat.

MATERIALS AND METHODS

Hard Red Winter wheat purchased locally at harvest and stored in bulk in a metal bin for 8 months, was passed through a shaker and fan-type seed cleaner to improve uniformity in kernel size and to remove most of the foreign material and broken kernels. Immediately after the wheat was cleaned and before it was treated, samples were taken to determine the moisture content, uniformity, and extent of insect damage.

Malathion emulsion spray, the standard treatment for comparison, was prepared from premium-grade, 57-percent malathion emulsifiable concentrate (EC) 0.6 kg/liter (5 lb AI/gal) and neutral distilled water for application of 10.42 p/m with 473 ml (1 pt) EC/27.3 metric tons. The malathion-Kenite 2-I dust was formulated to deposit 473 ml (1 pt) of 57 percent malathion EC in 27.2 kg (60 lb) of Kenite 2-I dust/27.3 metric tons (1,000 bu) of wheat.

Pirimiphos-methyl EC (formulation JF2764) 499.7 g/l (4.17 lb AI/gal) at 7.8 p/m AI, chlorpyrifos-methyl EC (formulation M-3721) 239.7 g/l (2 lb AI/gal) at 6.3 p/m AI, and fenitrothion (also known as Accothion and Sumithion) EC 0.96 kg AI/l (8 lb AI/gal) at 8.3 p/m AI were applied as emulsion sprays.

All emulsion sprays were applied with an ultra-low-volume atomizing spray assembly by siphon movement through a Spraying System Co. 1/4-in JN atomizing nozzle block at 4.5 kg/6.5 cm² (10 lb/in²) air movement at the rate of 18.9 liters (5 gal) finished emulsion spray/27.3 metric tons grain. The block was fitted with No. 2050 fluid and No. 70 air nozzles to deliver a cone-shaped spray. All spray materials were applied to 54.4 kg (2 bu) wheat through an aperture in the lid of 208 (55 gal) steel mixing barrels that rotated at 16 r/m on an electric barrel roller. The required amount of dust

was added to 54.4 kg wheat as it was poured into the barrel. The treated wheat was mixed for 15 min, and immediately after two lots were treated, the 108.8 kg (4 bu) of treated wheat was placed in 0.14 m³ (5 ft³) uncovered fiber drums for storage.

The grain was leveled to within 5 cm (2 in) below the top of the drums to provide equal areas for insect entry. There were four replications of each treatment with untreated check bins. The bins were stored in 5.2 × 6.7-m (17 × 22 ft) room at 26.7 ± 1.1°C and relative humidity (RH) 58 ± 5 percent. The treatments were placed in the bins in a selective randomized arrangement to locate

each treatment in four widely separated areas of the room. The temperature and RH in the infestation room favored insect development throughout storage. Major insect releases, each of about 6,400 rice weevils, 3,200 confused flour beetles and 3,200 red flour beetles, were made in the storage room 15, 40, 70, 135, 190, 230, 280, and 315 days after the test was started. Flat grain beetles, *Cryptolestes* spp., saw-toothed grain beetles, *Oryzaephilus surinamensis* (L.), and merchant grain beetles, *O. mercator* (Fauvel), were released by emergence from culture jars maintained in the storage room from 3 to 11 months of storage.

SAMPLING

Temperatures were taken with mercury thermometers from near the center of the grain mass in each bin at weekly intervals after treatment. Samples of wheat were probed from each bin with a nonpartitioned grain trier after 3, 6, 9, and 12 months' storage. The trier (probe) was inserted vertically near the center of each bin and about 6 cm (2 in) from the bin wall in each of the four quadrants until the required amount of grain, 3,000 g, was obtained for detailed studies of insect populations, test weights, kernel damage, and so forth. Additional probings were made for residue samples and other studies when required. The insects were immediately screened out of the probed sample and were counted for an estimate of the population in each bin. The fine dusts from the samples were returned for a thorough mixing with the parent samples. All grain samples were subjected to a temperature of about -30°C for 10 days to kill all hidden insect infestation. Before testing, these samples were held at 26°C and 60 percent RH for 48 hr following removal from the deep freeze to allow for moisture and temperature equilibration.

The 250-g subsamples of wheat were placed in 473-ml glass mason jars for toxicity tests. About 50 adult rice weevils, red and confused flour beetles, and lesser grain borers were each placed in separate jars for the bioassay tests. The live and dead insects were removed and counted 21 days later. After the mortality counts, all fine dusts and screenings were returned to the respective jars of wheat. The subsamples were held for an additional 42 days for F₁ progeny emergence of rice weevils, 49 days for red flour beetle and lesser grain borer, and 56 days for confused flour beetle emergence.

Following the F₁ progeny counts, all samples were retained for a 120-day visual assessment of damage of the developing infestations if any became established.

Bins were tightly covered during the probings to prevent insect migration from a bin being probed into the surrounding bins. The covers were removed 4 hours after sampling.

As a direct test for the acceptance or avoidance of the wheat treated with the different insecticidal formulations, about 250 rice weevil adults were released in multichoice food preference or selection chambers. In each chamber, 237 ml cardboard cartons, each filled with about 200 g of wheat from one of the different treatments and from the untreated check, were exposed to about 250 rice weevils released in the center depression. The rice weevils were allowed 24 hr to enter, leave, or remain in the cartons of treated wheat. At that time, the weevils were sifted from the wheat for counting. Each test was replicated five times. The grain for these studies was sifted and cleaned of all insects and insect frass and dusts before use; however, the fine dusts from the samples of wheat treated with the M + K dust formulation were returned to the parent sample for a thorough remixing of the dust and wheat before testing.

Repellency tests were conducted with replicated samples from all bins 7 days, 3, 6, 9, and 12 months after treatment. The treated grain and untreated check lots, which were handled in the same manner as in the food selection studies, were compared with untreated, uninfested source wheat. Five 237-ml cartons of treated wheat from a bin and five of untreated source wheat were alternated in the apparatus. About 500 rice weevil adults, 14 days old,

were liberated in the depressed release area located in the center of the chamber to scatter over the dispersal plane. The insects were given a 24-hr opportunity to choose from among the cartons of treated and untreated source wheat. Following the dispersal period, the weevils were sifted from the wheat for counting.

At the end of the 12-month storage, additional 3,000-g samples were probed from each bin. These

samples were screened over a 10-mesh screen to remove the kernel bits, insects, frass, and dusts. The screenings were sifted over a No. 25 sieve to separate the insect frass and other dusts from the insects and parts of kernels. The frass and dusts were weighed to estimate the insect damage to the grain. These fine siftings were remixed with the sifted grain and were stored in screen-covered 3.8-liter glass jars for 70 days to observe insect development and emergence.

RESULTS

As there was only one dosage of each formulation, the rates are not shown in the tables. They were as follows: 10.4 p/m malathion, 8.3 p/m fenitrothion, 7.8 p/m pirimiphos-methyl, 6.3 p/m chlorpyrifos-methyl, and 10.4 p/m malathion in the M + K dust formulation.

Grain Temperatures

Slight elevations in grain temperatures were noted in bins of untreated wheat during the latter part of the second month (table 1). Insect activity caused the temperatures to rise in all untreated bins until the fifth month. Thereafter the temperature in these bins gradually decreased. No marked temperature rise occurred in any of the bins treated with the different formulations during the first 12 months of storage, although slight elevations occurred in the bins filled with malathion (emulsion) treated wheat.

Moisture Content

The moisture content of the bin samples was determined on a Steinlite RCT-B moisture tester. The moisture content of the wheat had equilibrated

at 12.3 percent when the treatments were started. For the greater part of the storage, the treated lots remained near this level, except for the last 4 months when the room heating units were in constant operation (table 2). The moisture content in the untreated check bins remained near the initial content because of heavy insect activity throughout the test.

Residues

Table 3 shows the average residues found on the wheat during the 12-month storage. Samples taken 24 hr after treatment revealed that initial deposits of 83.3 percent pirimiphos-methyl, 82.7 percent malathion (emulsion), 95.2 percent chlorpyrifos-methyl, 74.7 percent fenitrothion, and 78.8 percent malathion (dust) were present on the wheat. Malathion, chlorpyrifos-methyl, and fenitrothion residues degraded gradually at about the same rate, but malathion on wheat residues from the dust formulation degraded more slowly than on wheat treated with the emulsion. Pirimiphos-methyl residues degraded in a somewhat erratic pattern; however, these data indicate that the pirimiphos-methyl residues are persistent in bulk grain storage

TABLE 1.—Average grain mass C° temperatures of insecticide-treated wheat during 12 months' storage

| Insecticide | Months of storage — | | | | | | | | | | | |
|---------------------------|---------------------|------|------|------|------|------|------|------|------|------|------|------|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| Sprays: | | | | | | | | | | | | |
| Malathion | 25.5 | 25.6 | 25.6 | 25.7 | 25.6 | 25.6 | 26.0 | 25.7 | 25.8 | 26.7 | 26.4 | 27.8 |
| Pirimiphos-methyl | 25.5 | 25.6 | 25.7 | 25.6 | 25.4 | 25.8 | 25.8 | 25.4 | 25.5 | 26.0 | 26.0 | 26.2 |
| Fenitrothion | 25.5 | 25.6 | 25.6 | 25.6 | 25.4 | 25.6 | 25.7 | 25.6 | 25.4 | 26.1 | 26.2 | 26.3 |
| Chlorpyrifos-methyl | 25.5 | 25.5 | 25.6 | 25.6 | 25.4 | 25.8 | 25.8 | 25.5 | 25.5 | 26.0 | 25.9 | 26.3 |
| Dusts: | | | | | | | | | | | | |
| M + K | 25.6 | 25.8 | 25.8 | 25.7 | 25.4 | 25.4 | 25.9 | 25.3 | 25.4 | 26.2 | 26.0 | 26.3 |
| Untreated: | | | | | | | | | | | | |
| Check | 25.3 | 26.0 | 29.7 | 31.7 | 33.0 | 32.6 | 32.0 | 31.2 | 30.1 | 29.8 | 29.4 | 29.4 |

because 83.1 percent of the initial deposit remained on the wheat after 12 months.

Insect Populations

Large numbers of insects were observed moving about in all areas of the storage room continuously after the first introduction of the rice weevils and flour beetles. Increased activity occurred when insect progeny began emerging from the check bins after about 70 days' storage.

The numbers of live adult insects that were recovered from the 3,000 g of wheat (probed samples) from each bin taken after 3, 6, 9, and 12 months indicated the relative active populations within the bins during storage (table 4). Small numbers of live insects were found in all bins of wheat treated with pirimiphos-methyl. Wheat treated with the malathion emulsion supported large numbers of adult insects after 9 months'

TABLE 2.—Average (percentage) moisture content of insecticide-treated wheat during 12 months' storage

| Insecticide | Before treat- ment | Months of storage | | | | |
|------------------------|--------------------------|-------------------|------|------|------|------|
| | | 1 | 3 | 6 | 9 | 12 |
| Sprays: | | | | | | |
| Malathion | 12.3 | 12.1 | 12.1 | 12.2 | 11.8 | 11.9 |
| Pirimiphos-methyl | 12.3 | 12.0 | 12.0 | 12.1 | 11.6 | 11.8 |
| Fenitrothion | 12.3 | 12.1 | 12.1 | 12.3 | 11.7 | 11.8 |
| Chlorpyrifos-methyl .. | 12.3 | 12.1 | 12.2 | 12.3 | 11.9 | 11.9 |
| Dusts: | | | | | | |
| M + K | 12.2 | 12.1 | 12.1 | 12.1 | 11.4 | 11.5 |
| Untreated: | | | | | | |
| Check | 12.4 | 12.3 | 12.3 | 12.4 | 12.2 | 12.3 |

TABLE 3.—Average residues in parts per million on Hard Winter wheat stored in small bins

| Insecticide | Calculated dose | Hours 24 | Months of storage | | | | | | | |
|-------------------------------|-----------------|-------------|-------------------|-----|-----|-----|-----|-----|-----|--|
| | | | 1 | 2 | 3 | 4 | 6 | 9 | 12 | |
| Emulsions: | | | | | | | | | | |
| Malathion | 10.4 | 8.6 | 5.2 | 4.5 | 3.8 | 3.5 | 3.0 | 2.0 | 1.4 | |
| Primiphos- methyl | 7.8 | 6.5 | 6.1 | 6.3 | 5.6 | 5.9 | 6.2 | 5.9 | 5.4 | |
| Fenitrothion .. | 8.3 | 6.2 | 4.9 | 4.0 | 3.4 | 3.0 | 2.6 | 1.6 | 1.3 | |
| Chlorpyrifos- methyl | 6.3 | 6.0 | 4.4 | 4.0 | 3.8 | 3.3 | 2.7 | 1.8 | 1.6 | |
| Dusts: | | | | | | | | | | |
| M + K ¹ | 10.4 | 8.2 | 6.7 | 5.5 | 4.6 | 4.3 | 3.9 | 2.6 | 2.0 | |

¹ Malathion residue.

TABLE 4.—Number of live adult insects recovered from 3,000-gram probed sample of insecticide-treated wheat during 12 months' storage¹

| Insecticide | Live insects in samples taken after — | | | |
|---------------------------|---------------------------------------|----------|-----------|-----------|
| | Months | | | |
| | 3 | 6 | 9 | 12 |
| Sprays: | | | | |
| Malathion | No. 2.0 | No. 30.7 | No. 202.8 | No. 886.0 |
| Pirimiphos-methyl | 10.7 | 14.0 | 11.3 | 5.0 |
| Fenitrothion | 8.0 | 11.3 | 12.0 | 52.0 |
| Chlorpyrifos-methyl | 4.0 | 32.3 | 16.0 | 27.3 |
| Dust: | | | | |
| M + K | 1.3 | 8.7 | 7.3 | 4.0 |
| Untreated: | | | | |
| Check | 535.8 | 1,221.5 | 988.5 | 1,370.2 |

¹ Average of 4 replications.

storage. The fenitrothion and chlorpyrifos-methyl treatments also gave good protection; however, populations were on the increase in the bins of wheat treated with fenitrothion during the last month of storage. The M + K dust treatment was the most effective in suppressing live insect populations in the bins (this protection was still effective 18 months after treatment).

Insect Emergence

After removal of the live and dead insects from the 3,000-g samples taken after 12 months' storage, the sifted corn and dusts, which were weighed, were recombined and retained for 70 days for counts of the numbers of insects that developed in these terminal samples.

The emergence of insects during the 70 days indicated the extent of the self-contained infestations that had become established (table 5). The fewest insects emerged from samples treated with the M + K dust formulation, and these insects did not establish an indigenous infestations (table 6). Also, damage was minimal with only minor infestations being established in two samples each of wheat treated with pirimiphos-methyl and chlorpyrifos-methyl. Damaging infestations of rice weevils, *Tribolium* spp., *Cryptolestes* spp., and *Oryzaephilus* spp., were established in all jars of wheat treated with fenitrothion and malathion.

Insect Damage

Assessments of insect damage to the wheat during 12 months' storage included the amounts of

TABLE 5.—*Number of emerging live adult insects recovered from 3,000-gram samples of insecticide-treated wheat after 12 months' storage*¹

| Insecticide | Rice weevils | <i>Tribolium</i> spp. | <i>Cryptolestes</i> spp. | <i>Oryzaephilus</i> spp. | Others | Total |
|---------------------------|--------------|-----------------------|--------------------------|--------------------------|--------|---------|
| Sprays: | | | | | | |
| Malathion | 602.8 | 116.8 | 207.8 | 69.5 | 208.8 | 1,205.3 |
| Pirimiphos-methyl | 56.3 | 14.8 | 16.8 | 0 | 79.5 | 167.8 |
| Fenitrothion ... | 417.5 | 141.3 | 346.5 | 72.3 | 403.0 | 1,380.3 |
| Chlorpyrifos-methyl | 60.5 | 20.3 | 20.0 | 0 | 77.5 | 178.3 |
| Dust: | | | | | | |
| M + K | 19.3 | 3.0 | 0 | 0 | 0 | 22.3 |
| Untreated: | | | | | | |
| Check | 513.0 | 186.5 | 404.2 | 162.5 | 216.0 | 1,482.3 |

¹ Samples were held for 70 days for emergence at end of 12 months' storage. Average of 4 replications.

TABLE 6.—*Ratings of visible damage by insects developing in samples of insecticide-treated wheat taken after 12 months' storage*¹

| Insecticide | Rating by replication number ² | | | | |
|---------------------------|---|---|---|---|---------|
| | 1 | 2 | 3 | 4 | Average |
| Sprays: | | | | | |
| Malathion | 3 | 4 | 4 | 3 | 3.5 |
| Pirimiphos-methyl | 0 | 1 | 1 | 0 | .5 |
| Fenitrothion | 4 | 3 | 3 | 3 | 3.3 |
| Chlorpyrifos-methyl | 0 | 0 | 1 | 1 | .5 |
| Dusts: | | | | | |
| M + K | 0 | 0 | 0 | 0 | 0 |
| Untreated: | | | | | |
| Check | 5 | 5 | 5 | 5 | 5 |

¹ Reading made 120 days after 12-month sampling.

² Damage rating code: 0 = no visible infestation; 1 = slight damage as evidenced by a few insects and a small amount of insect frass; 2, 3, and 4 = ascending numbers of insects and corresponding amounts of insect frass; 5 = large infestation with great amounts of insect frass and spoilage of grain.

insect frass in terminal samples, losses in test weight, percentages of kernels damaged by insects, and kernel weight losses. Weights of fine dusts, primarily insect frass, sifted from the samples taken after 12 months' storage indicated the amount of damage from insect feeding during storage (table 7). The small amounts of dusts recovered from the bins with the insecticidal treatments suggested minor insect damage to the wheat

kernels when compared to the large amount of insect frass from the untreated check bins. About three times as much dust was recovered from wheat treated with the standard malathion emulsion treatment than from all other formulations.

Changes in the test weight of wheat from the different treatments are shown in table 8. An average loss of 2.16 lb/bu was found in the bins of wheat treated with the standard dosage of malathion; however, little test weight loss occurred to wheat treated with pirimiphos-methyl, chlorpyrifos-methyl, and fenitrothion. No appreciable loss occurred during the 12 months in wheat storage treated with the M + K dust, but the application of the dust caused an immediate loss of 5.04 lb/bu of wheat because the adherence of the dust to the kernels affected the flowability and settling quali-

TABLE 7.—*Grams of insect frass per 3,000-gram sample of wheat 12 months after treatment*¹

| Insecticide | Average | Range |
|---------------------------|---------|-------------|
| Sprays: | | |
| Malathion | 14.3 | 9.9-19.2 |
| Pirimiphos-methyl | 4.7 | 3.9-5.7 |
| Fenitrothion | 6.1 | 4.2-8.1 |
| Chlorpyrifos-methyl | 5.1 | 4.1-5.9 |
| Dusts: | | |
| M + K | 4.1 | 3.5-4.9 |
| Untreated: | | |
| Check | 262.2 | 207.4-316.4 |

¹ Average of 4 samples per treatment.

TABLE 8.—*Average test weight (pounds) per bushel of wheat at given intervals during 12 months' storage*¹

| Insecticide | Immediately after treatment | Months of storage | | | | Loss during storage | |
|-------------------------------|-----------------------------------|-------------------|-------|-------|-------|---------------------------|-------|
| | | 3 | 6 | 9 | 12 | | |
| Sprays: | | | | | | | |
| Malathion ... | 63.20 | 63.34 | 62.86 | 61.90 | 61.04 | — | 2.16 |
| Pirimiphos- methyl | 63.45 | 63.42 | 63.14 | 63.04 | 63.09 | — | .36 |
| Fenitrothion . | 63.45 | 63.60 | 63.32 | 63.40 | 62.68 | — | .77 |
| Chlorpyrifos- methyl | 63.50 | 63.36 | 63.20 | 63.00 | 63.00 | — | .36 |
| Dust: | | | | | | | |
| M + K | 58.28 | 57.98 | 58.17 | 58.04 | 58.20 | — | .08 |
| Untreated: | | | | | | | |
| Check | 63.30 | 62.17 | 55.90 | 52.68 | 52.04 | — | 11.26 |

¹ Average initial test weight was 63.32 pounds per bushel before insecticides were applied.

² The initial loss of 5.04 pounds during treatment was caused by the addition of the diatomaceous earth.

TABLE 9.—Average percentage of kernels showing insect feeding and the calculated weight loss in samples of insecticide-treated wheat during 12 months' storage¹

| Insecticide | Months of storage | | | | Percent weight loss |
|---------------------------|-------------------|------|------|------|---------------------|
| | 3 | 6 | 9 | 12 | |
| Sprays: | | | | | |
| Malathion | 0.3 | 1.3 | 4.3 | 7.3 | 2.14 |
| Pirimiphos-methyl | 0 | 1.3 | 1.7 | 2.7 | .60 |
| Fenitrothion | 0 | 1.3 | 2.9 | 5.3 | 1.36 |
| Chlorpyrifos-methyl | 0 | 1.2 | 2.0 | 2.9 | .70 |
| Dust: | | | | | |
| M + K | 0 | 1.0 | 1.7 | 2.1 | .34 |
| Untreated: | | | | | |
| Check | 7.3 | 30.3 | 52.1 | 73.5 | 27.48 |

¹ No stored-product insect damage to kernels before treatment.

ties. The untreated wheat lost about 17.85 percent of its original weight.

Samples were periodically examined to determine the percent of kernels damaged by insect feeding. At the termination of the test, 1,000 kernels from each bin were examined and weighed to determine the amount of kernel weight loss from insect feeding. The weights of undamaged kernels averaged 0.0286 g, but the weights of damaged kernels varied greatly. Damage was heavy in the untreated wheat (table 9). In comparison, damage was relatively light in wheat with the standard malathion treatment as only 7.3 percent of the kernels showed insect feeding. Wheat treated with fenitrothion, pirimiphos-methyl, chlorpyrifos-methyl, and the M + K dust had only 5.3, 2.7, 2.9, and 2.1 percent of the kernels damaged, respectively, and little kernel weight loss occurred. Samples of kernels from the untreated check bins contained many heavily damaged kernels, which were broken up. Usually these kernels passed through the screen during removal of insects and dusts by the screening process. Consequently, losses to heavily damaged kernels are sometimes not recorded when determining the calculated weight loss to the kernels from insect feeding.

Food Selection Studies

Competitive multichoice offerings of samples from different treatments to 14-day-old rice weevil adults showed that the wheat covered with the M + K dust was definitely avoided, but none of the other insecticidal formulations affected the ac-

TABLE 10.—Response of rice weevils to insecticide-treated and untreated wheat in food selection tests¹

| Insecticide | Percentage of weevils that entered samples after storage period of — | | | | | |
|---------------------|--|---------|----------|----------|----------|-----------|
| | 7 days | 1 month | 3 months | 6 months | 9 months | 12 months |
| Sprays: | | | | | | |
| Malathion | 19.40 | 20.25 | 20.03 | 18.08 | 20.12 | 19.51 |
| Pirimiphos-methyl | 18.82 | 20.00 | 19.30 | 18.19 | 20.50 | 20.31 |
| Fenitrothion | 19.60 | 20.70 | 20.20 | 21.11 | 19.60 | 20.20 |
| Chlorpyrifos-methyl | 19.77 | 18.35 | 18.00 | 18.64 | 19.98 | 20.21 |
| Dust: | | | | | | |
| M + K | 2.55 | 2.80 | 4.30 | 3.17 | 3.00 | 3.44 |
| Untreated: | | | | | | |
| Check | 20.04 | 18.60 | 18.17 | 20.81 | 16.81 | 16.34 |

¹ Average of 5 replications per sampling period per treatment.

ceptability of the wheat (table 10). From 97.82 to 100 percent of the weevils released in the chambers entered the cartons. Heavily damaged wheat from the untreated check lots was less acceptable during the latter part of the test than during the first part of the storage.

Repellency Tests

Repellency tests were conducted with replicated samples 7 days, 3, 6, 9, and 12 months after treatment. Five replications of grain composited from

TABLE 11.—Percentage repellency of treated and untreated wheat to rice weevil adults

| Insecticide | Repellency after interval of ¹ | | | | |
|---------------------|---|----------|----------|----------|-----------|
| | 7 days | 3 months | 6 months | 9 months | 12 months |
| Sprays: | | | | | |
| Malathion | 1.00 | — 4.40 | 1.60 | 2.05 | — 0.40 |
| Pirimiphos-methyl | 1.40 | — 9.45 | 7.59 | — 7.02 | 7.54 |
| Fenitrothion | 3.98 | 4.59 | — 7.91 | 3.98 | 4.40 |
| Chlorpyrifos-methyl | 1.02 | .97 | .93 | — 1.10 | — 3.62 |
| Dusts: | | | | | |
| M + K | 62.00 | 70.63 | 78.80 | 69.76 | 76.28 |
| Untreated: | | | | | |
| Check | — .40 | — .39 | 2.38 | 10.54 | 9.92 |

¹ Equation for repellency: $100 - (T \div \frac{U+T}{2}) \times 100$

where *U* is the number of insects in the untreated wheat and *T*, the number in the treated wheat.

TABLE 12.—Average percent mortality of adult insects after 21 days exposure to insecticide-treated wheat with subsequent emergence of F_1 progeny

| Insecticide | Period between treatment and infestation of wheat | | | | | | | | | | | |
|-------------------------------------|---|---------|---------|-----------|---------|---------|-----------|---------|---------|-----------|---------|---------|
| | 3 months | | | 6 months | | | 9 months | | | 12 months | | |
| | Mortality | Progeny | | Mortality | Progeny | | Mortality | Progeny | | Mortality | Progeny | |
| | | Average | Dead | | Average | Dead | | Average | Dead | | Average | Dead |
| | Percent | Number | Percent | Percent | Number | Percent | Percent | Number | Percent | Percent | Number | Percent |
| RICE WEEVILS ¹ | | | | | | | | | | | | |
| Sprays: | | | | | | | | | | | | |
| Malathion | 100.0 | 10.3 | 100.0 | 100.0 | 43.3 | 98.6 | 99.4 | 148.0 | 39.2 | 86.7 | 283.0 | 8.7 |
| Pirimiphos-methyl .. | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 4.0 | 100.0 | 100.0 | 12.0 | 100.0 |
| Fenitrothion | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 4.0 | 92.3 | 90.0 | 57.1 | 61.2 |
| Chlorpyrifos-methyl | 100.0 | 0 | — | | | | 100.0 | 16.2 | 100.0 | 100.0 | 22.0 | 95.5 |
| Dust: | | | | | | | | | | | | |
| M + K | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 11.0 | 100.0 |
| Untreated: | | | | | | | | | | | | |
| Check | 0 | 1,245.0 | .1 | 0 | 1,052.0 | 0 | 0 | 648.1 | 1.2 | 2.2 | 791.3 | 1.8 |
| CONFUSED FLOUR BEETLES ² | | | | | | | | | | | | |
| Sprays: | | | | | | | | | | | | |
| Malathion | 73.5 | 5.0 | 80.0 | 58.6 | 4.0 | 0 | 12.3 | 7.6 | 0 | 4.5 | 13.0 | 0 |
| Pirimiphos-methyl .. | 99.4 | 0 | — | 100.0 | 0 | — | 97.6 | 0 | — | 84.5 | 4.0 | 100.0 |
| Fenitrothion | 63.6 | 2.0 | 50.0 | 66.7 | 0 | — | 23.3 | 0 | — | 22.6 | 15.1 | 20.0 |
| Chlorpyrifos-methyl | 98.0 | 0 | — | 98.2 | 0 | — | 97.3 | 0 | — | 80.0 | 7.3 | 93.1 |
| Dust: | | | | | | | | | | | | |
| M + K | 98.7 | 0 | — | 100.0 | 0 | — | 89.8 | 0 | — | 71.8 | 0 | — |
| Untreated: | | | | | | | | | | | | |
| Check | 0 | 56.2 | 0 | 0 | 64.0 | 0 | .4 | 57.0 | 1.5 | 1.4 | 67.0 | .4 |
| RED FLOUR BEETLES ³ | | | | | | | | | | | | |
| Sprays: | | | | | | | | | | | | |
| Malathion | 80.0 | 0 | — | 76.0 | 0 | — | 46.4 | 4.0 | 100.0 | 7.0 | 23.2 | 0 |
| Pirimiphos-methyl .. | | | | | | | | | | | | |
| Fenitrothion | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 1.3 | 100.0 |
| Chlorpyrifos-methyl | 100.0 | 0 | — | 98.8 | 0 | — | 74.4 | 0 | — | 29.0 | 14.4 | 7.7 |
| Dust: | | | | | | | | | | | | |
| M + K | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 2.5 | 90.0 |
| Untreated: | | | | | | | | | | | | |
| Check | 0 | 54.2 | 0 | 1.0 | 47.3 | 1.3 | .5 | 86.0 | 0 | 1.4 | 68.0 | 0 |
| LESSER GRAIN BORER ¹ | | | | | | | | | | | | |
| Sprays: | | | | | | | | | | | | |
| Malathion | 93.9 | 0 | — | 75.6 | 5.2 | 25.2 | 31.1 | 44.3 | 22.8 | 16.0 | 59.6 | 17.3 |
| Pirimiphos-methyl .. | 91.0 | 0 | — | 79.2 | 4.0 | 37.5 | 45.1 | 33.0 | 25.0 | 33.2 | 76.0 | 26.3 |
| Fenitrothion | 91.1 | 0 | — | 67.9 | 11.3 | 24.4 | 32.0 | 47.8 | 28.2 | 19.0 | 67.8 | 25.8 |
| Chlorpyrifos-methyl | 95.3 | 0 | — | 92.0 | 0 | — | 79.5 | 9.5 | 60.5 | 49.3 | 16.0 | 46.9 |
| Dust: | | | | | | | | | | | | |
| M + K | 100.0 | 0 | — | 100.0 | 0 | — | 100.0 | 0 | — | 97.0 | 2.0 | 100.0 |
| Untreated: | | | | | | | | | | | | |
| Check | 0 | 765.2 | .2 | 0 | 650.3 | .8 | 0 | 548.2 | 1.7 | 0 | 887.0 | .6 |

¹ Counts of progeny were made 63 days after initial infestation.

² Counts of progeny were made 77 days after initial infestation.

³ Counts of progeny were made 70 days after initial infestation.

each bin treatment were compared with cleaned, untreated, and uninfested wheat from the source

lot used for all treatments. The M + K dust treatment imparted repellency to the wheat to rice

weevils, but the other treatments did not impart repellency nor add to the attractiveness of the wheat (table 11). Grain from the untreated check bins that was heavily damaged during the latter part of the storage period was avoided to some extent by rice weevils in the 9- and 12-month tests.

Toxicity Studies

Mortalities of the adult insects were determined by no-choice bioassays conducted with 21-day exposures to the insecticide-treated grain. Pirimiphos-methyl, chlorpyrifos-methyl, and the M + K dust gave complete kills of adult rice weevils in all samples for 12 months, and F₁ progenies that emerged were effectively controlled by the residues (table 12). The effectiveness of malathion and fenitrothion decreased during the last 3 months of the storage, but fenitrothion gave controls superior to malathion.

Pirimiphos-methyl and chlorpyrifos-methyl were effective against confused and red flour beetles during the 12 months' storage. The M + K dust, although not killing as many of the adults as these two insecticides, prevented F₁ progeny production. No damage occurred in the bioassay samples retained for assessment of progeny damage (table 13). Fenitrothion and malathion were not nearly as effective against the adult flour beetles, but progeny production was greatly suppressed.

The M + K dust was effective in bioassays with lesser grain borer adults and almost complete protection was afforded the wheat. Chlorpyrifos-methyl gave relatively good protection during the first part of the storage period, but its effectiveness gradually declined during the last months of storage. After 9 months' storage, samples of wheat from the other treatments suffered considerable damage by the lesser grain borers, and F₁ progeny established indigenous infestations in all samples.

Visual assessments of progeny damage resulting from the toxicity tests are shown in table 13. Wheat treated with the M + K dust was completely protected from all four test species. Pirimiphos-methyl and chlorpyrifos-methyl prevented the establishment of indigenous infestations by the confused and red flour beetles for 12 months, and only minor infestations of rice weevils were recorded. Chlorpyrifos-methyl gave complete protection from the lesser grain borer for 9 months, and progeny damage was minimal after the 12-month tests.

TABLE 13.—*Ratings of visible damage by insect progeny in samples of insecticide-treated wheat after toxicity tests*

| Insecticide | Damage observed 120 days after bioassay infestation of samples taken after a storage period of— ¹ | | | |
|------------------------------|--|------------------|------------------|------------------|
| | 3 months | 6 months | 9 months | 12 months |
| RICE WEEVIL | | | | |
| Sprays: | | | | |
| Malathion | 0 | 0 | 2.0 | 2.8 |
| Pirimiphos-methyl | 0 | 0 | 0 | .3 |
| Fenitrothion | 0 | 0 | 0 | 1.5 |
| Chlorpyrifos-methyl ... | 0 | 0 | 0 | .5 |
| Dusts: | | | | |
| M + K | 0 | 0 | 0 | 0 |
| Untreated: | | | | |
| Check | ² 5.0 | 5.0 | 5.0 | ² 5.0 |
| CONFUSED FLOUR BEETLE | | | | |
| Sprays: | | | | |
| Malathion | 0 | 0 | 0.8 | 1.0 |
| Pirimiphos-methyl | 0 | 0 | 0 | 0 |
| Fenitrothion | 0 | 0 | .4 | .4 |
| Chlorpyrifos-methyl ... | 0 | 0 | .2 | 0 |
| Dusts: | | | | |
| M + K | 0 | 0 | 0 | 0 |
| Untreated: | | | | |
| Check | 2.0 | 2.0 | 2.4 | 2.6 |
| RED FLOUR BEETLE | | | | |
| Sprays: | | | | |
| Malathion | 0 | 0 | 0 | 1.0 |
| Pirimiphos-methyl | 0 | 0 | 0 | 0 |
| Fenitrothion | 0 | 0 | 0 | .2 |
| Chlorpyrifos-methyl ... | 0 | 0 | 0 | 0 |
| Dusts: | | | | |
| M + K | 0 | 0 | 0 | 0 |
| Untreated: | | | | |
| Check | 1.2 | 2.0 | 2.0 | 2.5 |
| LESSER GRAIN BORER | | | | |
| Sprays: | | | | |
| Malathion | 0 | 0 | 1.0 | 3.0 |
| Pirimiphos-methyl | 0 | 0 | .5 | 2.0 |
| Fenitrothion | .5 | 1.0 | 1.0 | 4.5 |
| Chlorpyrifos-methyl ... | 0 | 0 | 0 | 1.0 |
| Dusts: | | | | |
| M + K | 0 | 0 | 0 | 0 |
| Untreated: | | | | |
| Check | ² 5.0 | ² 5.0 | ² 5.0 | ² 5.0 |

¹ Damage rating code: 0 = no visible infestation; 1 = slight damage as evidenced by a few insects and a small amount of insect frass; 2, 3, and 4 = ascending numbers of insects and corresponding amounts of insect frass; 5 = large infestation with great amounts of insect frass and/or spoilage of grain.

² Damage after 90 days.

CONCLUSIONS

The protectant properties of three candidate emulsifiable concentrates and one dust were compared with those of the standard malathion treatment. Results were remarkably consistent between replications of the individual treatments throughout the storage study, and there was good correlation among results of the different methods of evaluation of the effectiveness of the individual treatments.

The following conclusions were drawn from the results of the study:

1. The malathion-diatomaceous earth (M + K) dust was completely effective in toxicity tests with rice weevils, confused and red flour beetles, and lesser grain borers, and prevented the development of indigenous infestations.

2. Application of the M + K dust reduced the test weight of the wheat about 5 lb/bu, consequently lowering the commercial grade.

3. Pirimiphos-methyl was effective in toxicity tests with rice weevils and confused and red flour beetles, but lesser grain borers were somewhat tolerant to the killing action of its residues.

4. About 83.3 percent of the intended dosage of pirimiphos-methyl was recovered 24 hr after treatment. After 12 months' storage, 83.1 percent of the initial deposit remained on the wheat.

5. The treatment with pirimiphos-methyl prevented insect infestations in the bins and appreciable losses in test weight and damage to the wheat kernels from insect feeding.

6. Chlorpyrifos-methyl was effective in the tox-

icity tests and ranked next to the M + K dust in controlling the lesser grain borer.

7. Chlorpyrifos-methyl residues degraded gradually during the 12-month storage. About 95.2 percent of the intended dosage, found on the wheat 24 hr after treatment, degraded to about 25.4 percent during storage.

8. The chlorpyrifos-methyl treatment prevented the establishment of insect infestations in the wheat and prevented losses in test weight and damage to the kernels.

9. Fenitrothion treatment gave good protection for 12 months but did not prevent the establishment of an indigenous infestation of mixed species of insects during the last month of storage.

10. Fenitrothion and malathion residues degraded gradually at about the same rate over the 12-month storage.

11. Fenitrothion was generally more effective than malathion throughout the test period.

12. The M + K dust treatment imparted repellency to the wheat as food for rice weevils in both the food selection and repellency tests. Other treatments did not affect the acceptability of the wheat.

13. Malathion residues degraded more slowly on wheat treated with the dust formulation than on wheat treated with the emulsion.

14. The order of general effectiveness at the dosages applied was M + K dust > pirimiphos-methyl > chlorpyrifos-methyl > fenitrothion > malathion.

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